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1/77

# Request for grant of a patent

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13 AUG 2003

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Cardiff Road
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1. Your reference

RSJ07869GB

2. Patent application number
*(The Patent Office will fill in this part)*

0319043.6

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Anglo-European Design Engineers Limited
Warrens Cottage,
Bridge Street,
Wickham, Fareham,
Hants PO17 5JF
Great Britain

Patents ADP number *(if you know it)*

08692113001

If the applicant is a corporate body, give the country/state of its incorporation

Great Britain

4. Title of the invention

ELECTROCHEMICAL GAS SENSORS

5. Name of your agent *(if you have one)*

Gill Jennings & Every

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Broadgate House
7 Eldon Street
London
EC2M 7LH

Patents ADP number *(if you know it)*

745002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

| Country | Priority application number *(if you know it)* | Date of filing *(day / month / year)* |
|---|---|---|

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

| Number of earlier application | Date of filing *(day / month / year)* |
|---|---|

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

*a) any applicant named in part 3 is not an inventor, or*
*b) there is an inventor who is not named as an applicant, or*
*c) any named applicant is a corporate body.*
*See note (d))*

YES

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| Continuation sheets of this form | |
| Description | 14 |
| Claim(s) | 2 |
| Abstract | 0 |
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| Priority documents | |
| Translations of priority documents | |
| Statement of inventorship and right to grant of a patent *(Patents Form 7/77)* | |
| Request for preliminary examination and search *(Patents Form 9/77)* | |
| Request for substantive examination *(Patents Form 10/77)* | |
| Any other documents *(please specify)* | NO |

11. For the applicant
Gill Jennings & Every

I/We request the grant of a patent on the basis of this application.

Signature [signature] Date 13 August 2003

12. Name and daytime telephone number of person to contact in the United Kingdom

SKONE JAMES, Robert Edmund
020 7377 1377

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Figure 1(a)

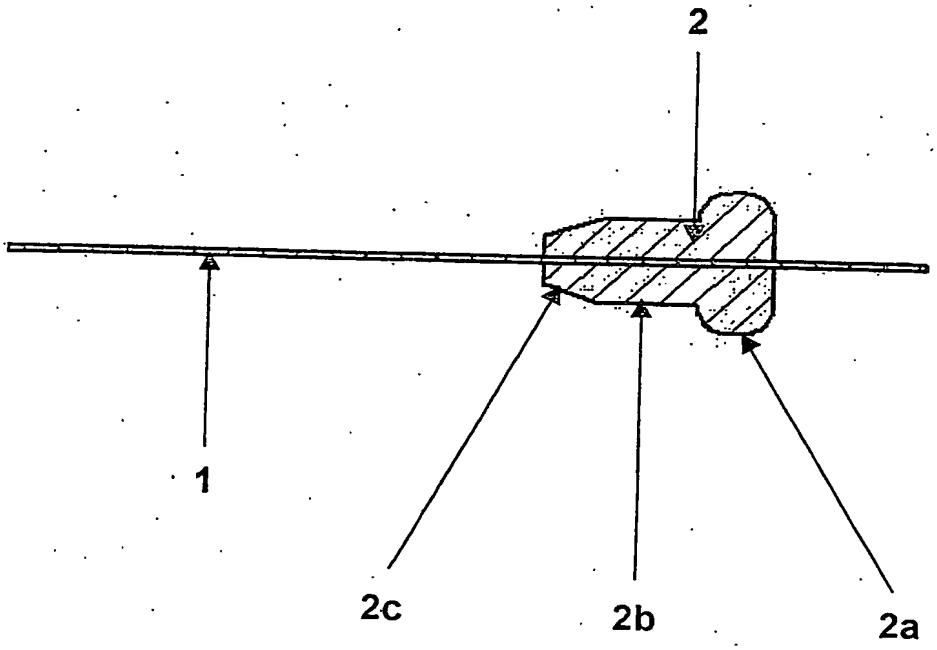



Figure 1(b)

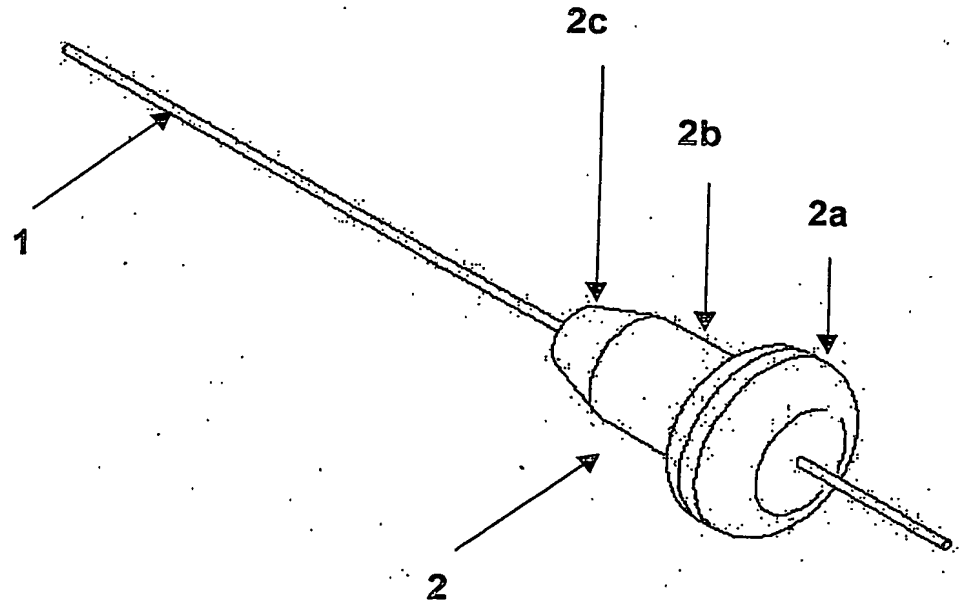


Figure 2

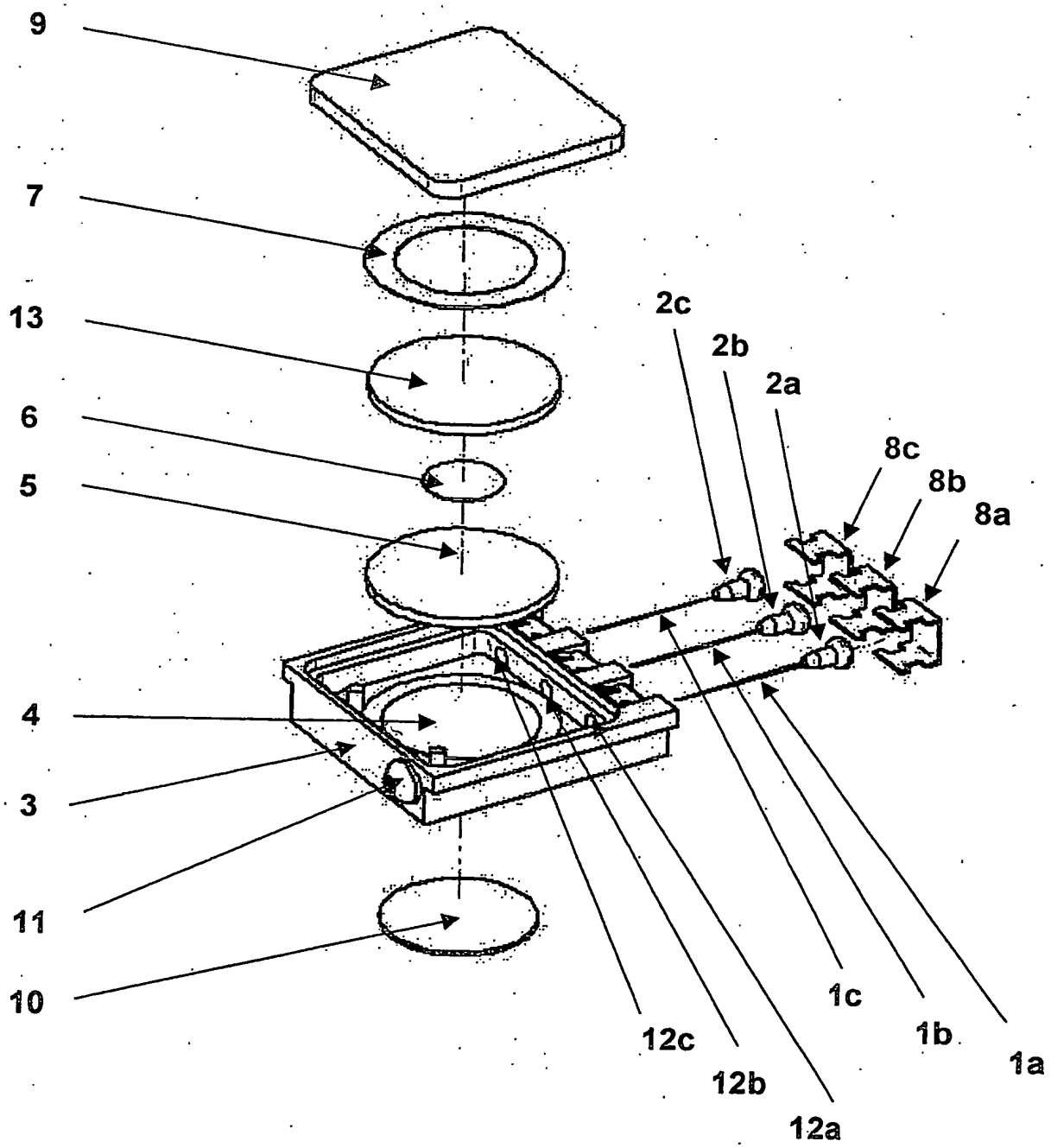
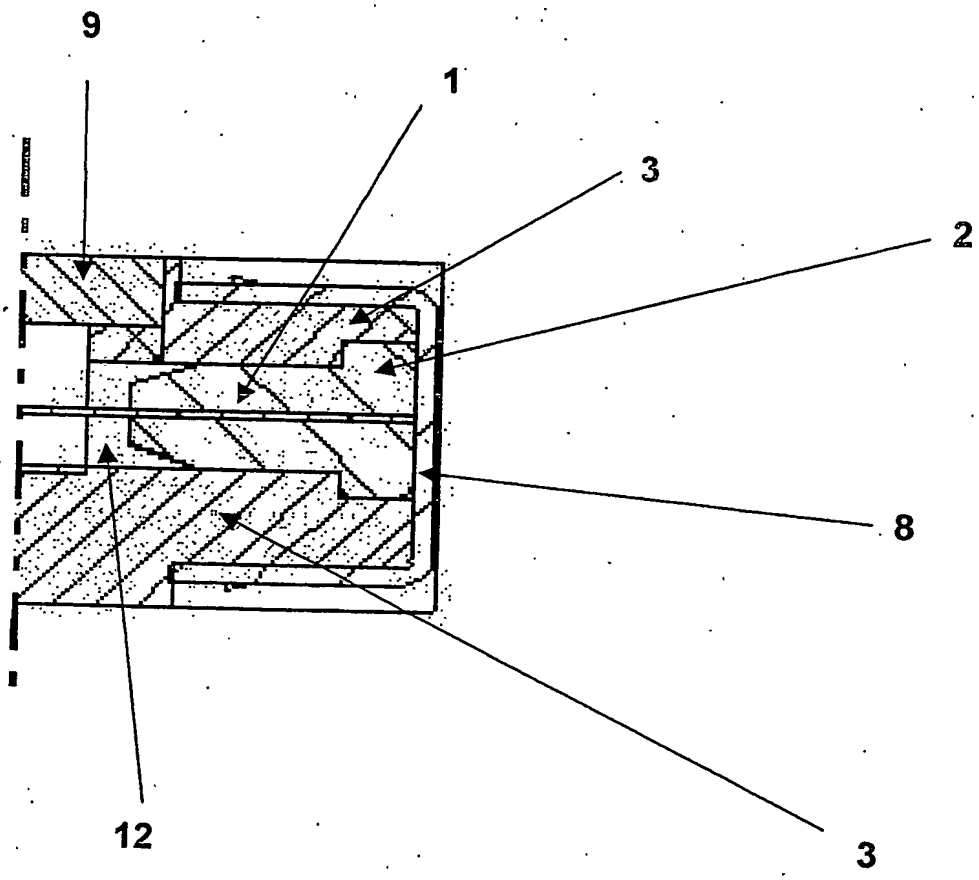


**Figure 3**



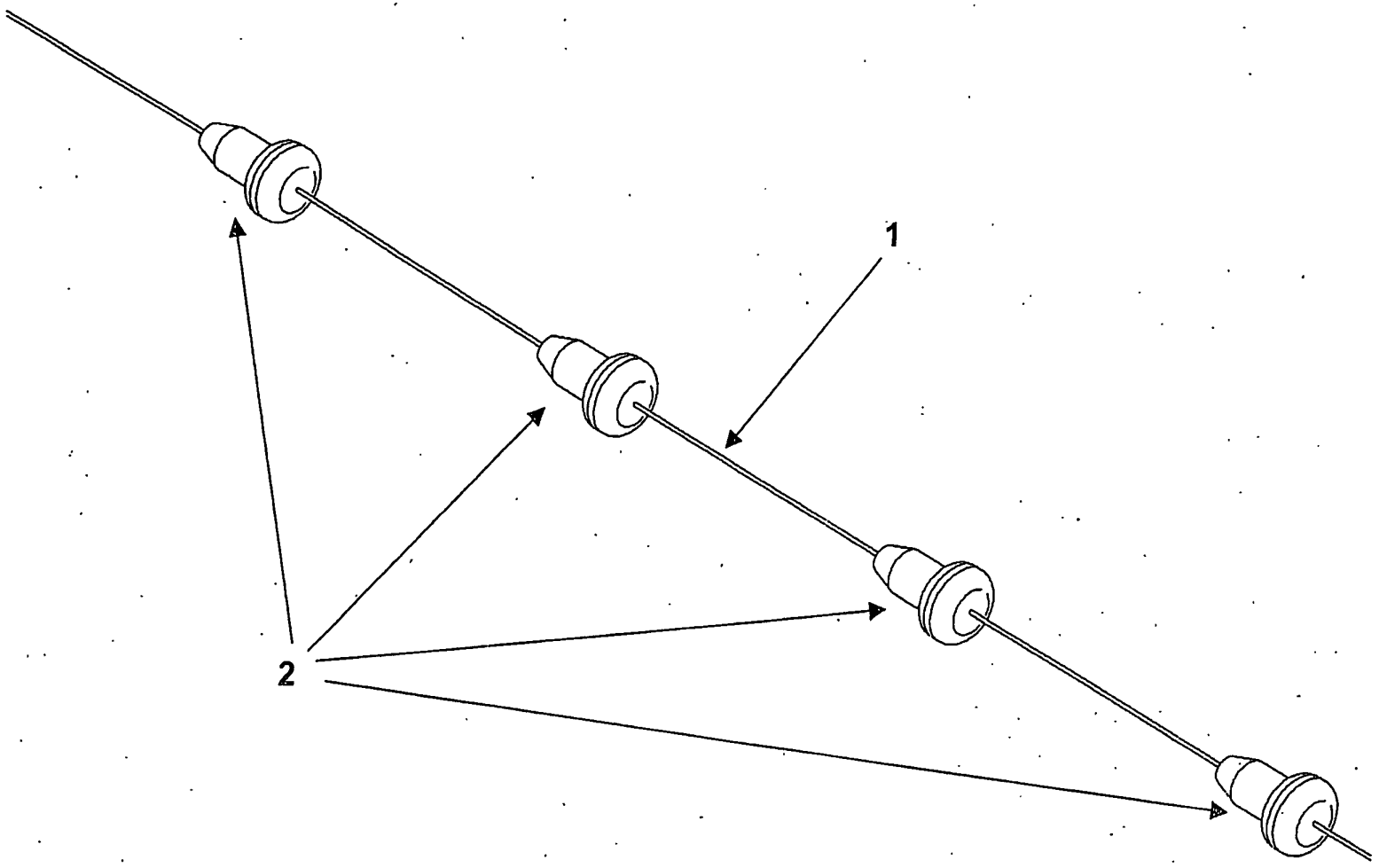



**Figure 4**

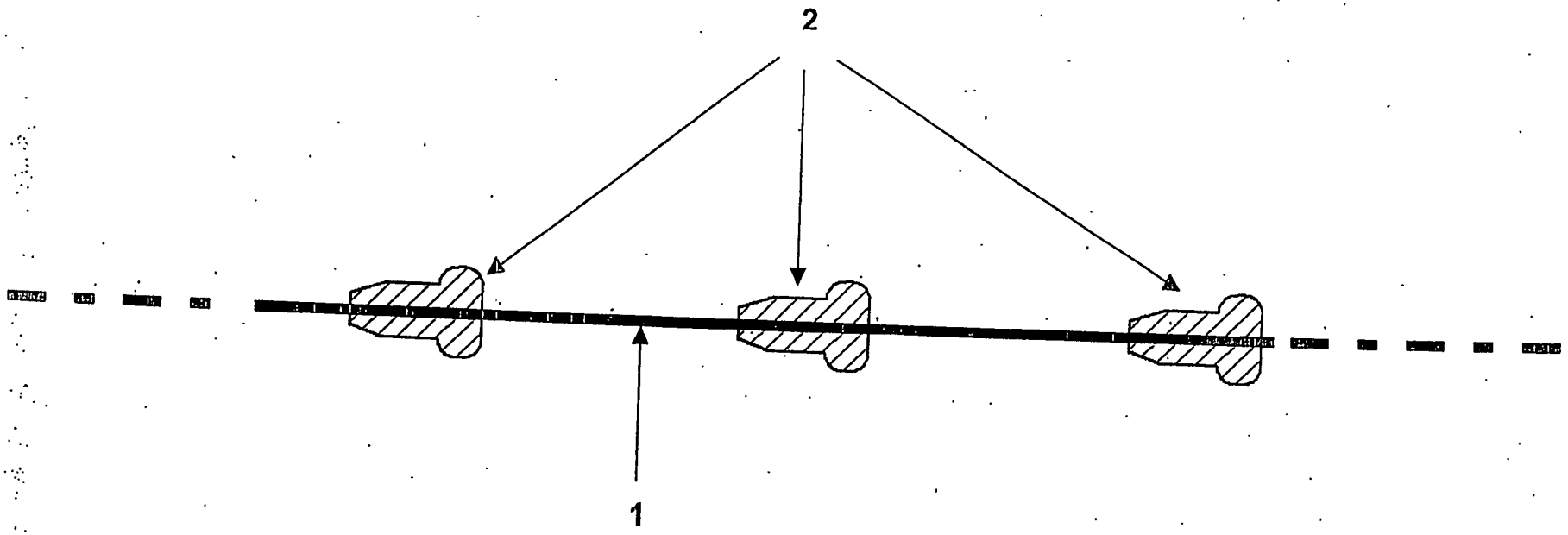
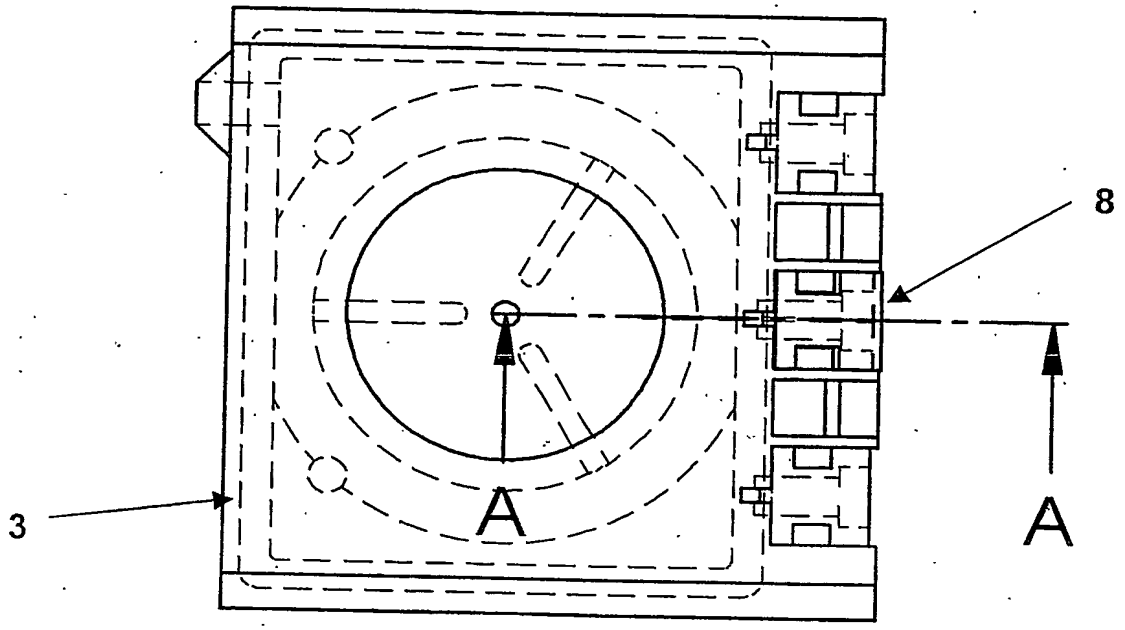
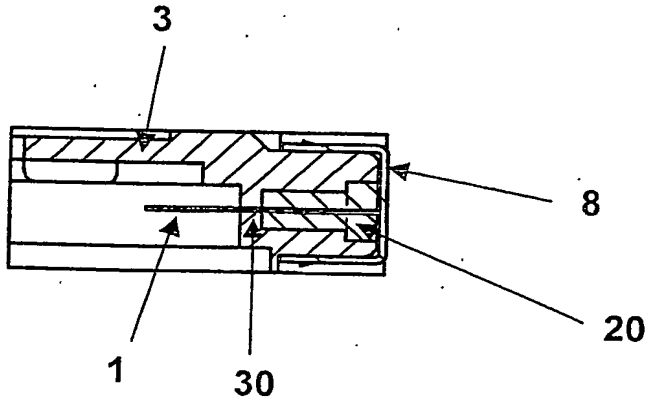



**Figure 5**

Figure 6




(A)




SECTION A-A

(B)

# ELECTROCHEMICAL GAS SENSORS

In electrochemical gas sensors based on liquid electrolytes and gas diffusion electrodes, it is necessary to provide a low resistance electrical contact to each electrode. This in turn connects to the external circuitry used to control and/or interrogate the device. Although a variety of materials may in principle be used to fabricate these `current collectors', it is essential that there is no corrosion reaction with the electrolyte. For this reason, in toxic sensors which contain extremely aggressive acidic electrolytes, it is most common to employ noble metal current collectors. Since such electrolytes must be contained within a defined envelope it is necessary for electrical connection to be made through a seal defining part of the envelope wall.

A number of approaches to meeting this requirement have been suggested, including the use of plastic sensor housings with conductive portions which press against peripheral regions of the electrodes, and the potting of electrode edges and emergent current collectors in epoxy or similar materials. However, neither offers a high performance, reliable and easily manufactured solution to the problem; a better approach is described in GB2094005. Here, the current collectors are small diameter wires or flattened ribbons of noble metals, interleaved with the gas diffusion electrodes and insulating separators in a stack arrangement which is wetted by electrolyte from a reservoir via a wick. The current collectors provide good contact across the electrode face, thereby ensuring efficient cell performance, and can emerge from the stack where the gas porous backing material of 2 or more of the electrodes is brought into contact in a peripheral annular zone. By arranging for adequate pressure to be applied in this region (for example via an O ring at the top of the stack which is compressed on assembly of the housing), an electrolyte-tight seal may be produced. The performance of

this seal relies on the ability of the electrode backing material (typically PTFE) to creep under pressure and so form itself around the current collector. Thus, gas is able to diffuse in to the active region, but electrolyte is contained by the hydrophobic properties of the electrode backing tapes and the annular seal.

The relatively fragile current collector is not a suitable means of making electromechanical contact to the sensor in a rugged industrial environment where repeated insertions into sockets may be required over the device life. It is therefore common to join the flexible current collector to a lower cost pin, pad or other conventional connection means in a region of the housing where no electrolyte contact will occur. A double skin sensor wall construction may also be employed to provide further protection of the current collector as it travels from the seal to the external connection means.

Commercial experience over many years has shown that current collector seals fabricated as described in GB2094005 are capable of high levels of performance, but has also highlighted a number of problems.

(1) The manufacturing process is difficult to automate due to the complexity of the assembly, especially in systems employing multiple electrodes and so requiring several current collectors. A number of steps must be completed simultaneously; (a) alignment of multiple current collector ribbons, (b) alignment of 2 or more electrode backing tapes, (c) ultrasonic or other closure of the outer sensor casing to produce adequate pressure in the seal area.

(2) There are difficulties in implementing such designs in sensors having significantly reduced dimensions due to manual handling complexities and the need for an adequate seal length (i.e.

the footprint of the annular seal zone needed to ensure integrity throughout the sensor life).

(3) The approach demands that the seal design perform a number of functions. It must retain an aggressive liquid electrolyte over a period of years whilst subject to significant fluctuations in temperature, humidity and other environmental factors. Simultaneously, it must not compromise the mechanical or electrical integrity of the current collectors passing through it. It must also maintain the relative positions of the electrodes and current collectors without allowing any movement which could compromise the cell performance in any orientation. Therefore, optimal performance in any one aspect is usually obtained at the expense of behaviour which is compromised in other respects.

Whilst rigid current collectors are not favoured in sensor designs of the type described in GB2094005, electrochemical gas sensor designs based around robust, inflexible current collectors are certainly feasible. These would normally rely on an O ring seal of the type described in WO-A-01/81911 to retain the electrolyte. However, when attempting to miniaturise sensors and use smaller pins and O rings, the seal length (i.e. the length of pin and sensor housing in contact with the inner and outer surfaces of the O ring) becomes rather small and this may compromise the overall integrity of the design. Furthermore, the features required in the housing in order to produce compression of the O ring occupy valuable space and further mitigate against attempts to miniaturise the arrangement. The stress which almost inevitably exists within the sensor housings in such circumstances is also a potential source of failure, particularly as the device experiences thermal transients during its life. These

problems are common to many types of gas sensor, and although some partial solutions have been proposed, difficulties remain. Such problems are greatly exacerbated when seeking to significantly reduce the size of sensors to meet the demands of instrument manufacturers.

In accordance with a first aspect of the present invention, we provide an electrochemical gas sensor having a housing in which is located sensing and counter electrodes in contact with a liquid electrolyte; and current collectors in contact with respective electrodes and extending through respective apertures in the housing, each aperture including a compliant seal which is resiliently urged against the current collector and the wall of the aperture to seal against the passage of electrolyte through the aperture.

This invention overcomes the problems of the prior art by avoiding any reliance on seals produced by the compression of gas diffusion electrode backing tapes, external potting material and the like. The invention allows the fabrication of electrochemical gas sensors with much smaller volumes than have been achieved in the past, for example as much as 75% less.

In some aspects, the compliant seal could be formed by back filling the aperture with a suitable thermoplastic elastomer and this will be described in more detail below. In the preferred examples, however, the compliant seal comprises an elastomeric grommet.

In our preferred design, the relatively fragile, flexible current collector passes through a central channel formed in a grommet. The grommet is typically initially a loose fit on the current collector although it could be a tight fit. A relatively long seal length is obtained by ensuring that the grommet length (e.g. 3mm) is large in comparison with both the diameter of the internal channel (e.g. 100 microns) and the external diameter (e.g. 1.2mm).

The grommet may be fabricated by a conventional injection moulding process, using one of a number of

thermoplastic elastomer (TPE) materials which provide the required combination of compliance and resistance to chemical attack by aggressive electrolytes. The material selected, in combination with the design tolerances, must also provide the required degree of matching between the expansion properties of the harder plastic sensor housing and the current collector across a range of environmental conditions, thereby ensuring the integrity of the seal throughout the sensor life.

The grommet may be designed to be an interference fit into a receiving aperture in the sensor housing and is typically made from a material whose compliance is significantly greater than that of the housing. If the receiving aperture diameter is slightly smaller than the relaxed diameter of the grommet, the compliant nature of the grommet allows insertion to take place without any damage to the current collector passing through the centre. Once insertion is complete, however, the elastomeric properties of the grommet produce significant sealing forces which act to force the housing-grommet and the grommet-current collector surfaces into intimate contact. These forces, applied along the long contact path, provide a high integrity seal against electrolyte leakage, even when using liquids which have the ability to `track' along small apertures.

The long sealing path may, if preferred, be used to create a number of separate seals along the length of the grommet. For example, by providing the cylindrical body of the grommet with a ridged structure, the sealing forces generated following insertion into the receiving aperture may be enhanced in a series of laterally spaced annular zones. Control of the grommet and housing dimensions and construction materials ensure that excessive forces, unsupportable by the current collector passing through the centre, are not produced. Such a ridged, or saw-tooth construction may also assist in the insertion of the grommet, as the tolerance demands upon the receiving

aperture are somewhat relaxed in comparison with the situation which exists when a smooth bore approach is employed.

Additional lateral compression of the grommet may also be used in order to generate the required sealing forces if the material used is appropriately compliant. For example, a lateral extension of the grommet may be compressed after insertion using a clip which locates over the external moulded sensor housing. This approach may be employed even if the receiving aperture is marginally larger than the grommet. Within the bounds of the overall approach described by the invention, the seal designer may make appropriate tradeoffs in the detailed specification.

The behaviour of the grommet may be contrasted with that of a conventional O ring. The latter is designed to produce a relatively high sealing force over a short lateral distance by compressing the compliant O ring between two comparatively rigid surfaces. The rather malleable nature of the current collectors preferably used in gas sensors of the type described here renders them unable to support the required forces without deformation and hence leakage. Indeed, such small diameter members are unlikely to offer great strength, irrespective of the material used in their construction. There are also difficulties inherent in producing compliant components with such small apertures in a low cost, reliable fashion. As a result, conventional O rings suitable for sealing such small diameter components are not generally available.

The insertion of the grommet into the preformed aperture in the rigid sensor housing not only provides an excellent seal against electrolyte leakage during use. It also holds the current collectors in position relative to the case so that they can be folded down into the electrode stack at appropriate stages during the assembly process. The base closure does not require any additional alignment of the current collectors, as is the case when the electrolyte seal is produced by pressure applied by the

overall housing closure. This improvement simplifies the sensor assembly, and, since automated insertion of the grommet/current collector assembly may be envisaged, removes one of the main obstacles which has historically prevented machine assembly of sensors of this type.

We have considered a number of methods whereby the current collector/grommet assembly may be fabricated as a subassembly ready for incorporation into sensors as described above.

(1) Notwithstanding the earlier comments regarding the difficulties of manufacturing small compliant O rings, it is certainly possible to design moulding processes which can produce components of the type required here. For example. a central mandrel may be inserted into the mould and then withdrawn afterwards in order to create the central aperture. There is also the possibility of drilling through a solid grommet in order to create the aperture. Mechanical drilling of compliant materials presents many difficulties, and laser drilling is probably a better option, although the high aspect ratio of the aperture may present problems. Whatever method is selected to preform the grommet, it is then necessary to thread the current collector through the central aperture so that the grommet provides an interference fit to the wire in the completed assembly. But since noble metal wires of diameters in the 100 micron range offer relatively little resistance to buckling stresses and the length of the aperture is of the order of mm, this process is difficult. Overall, therefore, although the production of grommets which are subsequently threaded with current collectors is possible, it does not

offer an optimum, simple low cost route to the production of this key assembly.

(2) A much preferred solution is to overmould the grommet directly on to the current collector, for example by injection moulding. Alternatively, a range of casting or 'dough moulding' methods may be employed. The required aperture dimensions are automatically provided and suitable choice of grommet material ensures that no damage occurs to the current collector during the process. A further advantage is that multiple grommets may be created at suitable spacings on a length of current collector in a single moulding process. In yet a further enhancement, we envisage repeating this process on a continuous length of current collector wire which is automatically incremented or indexed through the mould when it opens at the end of each run. Thus, one may produce a reel of current collector material with appropriately spaced grommets mounted thereon which can in turn be used to feed an automated assembly machine in the sensor production process.

(3) It would also be possible to overmould the current collector seal after the current collector has been located within the exit port of the sensor housing. The wire must be (at least) temporarily retained in position relative to the opening or port. Although accurate positioning of such a thin wire in a relatively large opening presents challenges, one favoured approach would be to overmould at least part of the relatively hard sensor casing around the current collector wire itself in a first shot. (This would not produce a satisfactory long term seal for the sensor due to the mismatch in properties between the current collector and the

casing - which the grommet is designed to accommodate - but would certainly provide good retention of the current collector whilst the remaining assembly processes took place.) Subsequently, the current collector seal would be formed by `backfilling' by a second shot of TPE. This technically sophisticated approach offers significant further reductions in labour required in the overall assembly process.

Taking into account the conflicting demands of lower cost, the desire for increased automation of sensor assembly and the practical difficulties of the various approaches, the method (2) is currently seen as the most favourable and the example described below is based on this approach.

The sealing approach described here, in which the current collector exits through the body of the sensor (and in which the compliant tape stack perimeters are not a primary source of electrolyte retention) overcomes problems which are particularly challenging in the case of miniaturised sensors. However, many of the advantages offered by this method are applicable to larger sensors and the ability to redesign such devices to incorporate these improvements is a further benefit offered by the invention.

The electromechanical termination of the current collector after it exits the seal may be achieved in a number of ways, drawing on the known methods of prior art. A favoured approach is to clamp or spot weld the current collector to a clip which locates on the external body of the sensor. A flat area on the clip may then act as a contact pad, mating with appropriate connector features in the instrument where the sensor is to be employed.

It will be appreciated that the invention is applicable to many types of sensor including toxic gas and oxygen sensors.

Some examples of electrochemical gas sensors and fabrication methods according to the present invention will now be described with reference to the accompanying drawings, in which:-

Figures 1a and 1b are a longitudinal section and isometric view respectively of a current collector and grommet assembly;

Figure 2 is an exploded view of a first example of a sensor;

Figure 3 is a cross-section through a grommet and current collector located in the sensor housing apparatus;

Figures 4 and 5 are a perspective view and cross-section respectively through a plurality of grommets threaded on a wire; and

Figures 6A and 6B are a plan and section on A-A respectively of a second example of a sensor.

Figures 1 and 2 show views of a 100micron diameter platinum (or nickel in the case of an oxygen sensor) current collector wire 1 overmoulded with a grommet 2 made from a thermoplastic elastomer (Santoprene 64) having a 64 Shore A hardness rating. The overmoulding was performed in a specially designed mould tool which simultaneously produced four such grommets along the wire 1 on a pitch of 16mm. The current collector wire 1 may be fed into the mould tool under tension from a continuous reel arrangement, although in this case a length was cut from the reel and tensioned using appropriate weights. After removal from the mould tool, the assembly was diced to produce 4 identical overmoulded current collectors of the type shown in Figure 1. The overall length of the grommet is 3.0mm, comprising a bulbous extension 2a (1.0mm long x 2.0mm diameter); a cylindrical body 2b (1.24mm long x 1.2mm diameter); and a tapered front section 2c (0.76mm long, narrowing to 0.7mm diameter).

The use of these current collectors in the assembly of a 3 electrode Carbon Monoxide sensor is shown in Figure 2. A sensing electrode 4 is of conventional construction and

comprises a gas diffusion catalyst (typically high surface area platinum) pressed on to a 12mm diameter hydrophobic gas permeable backing tape, typically of PTFE. This is heat sealed into a sensor body 3 around the perimeter of the backing tape so that the electrocatalyst is uppermost in the orientation shown in Figure 2. If required, filter cloth(s) or suitably packaged filter powder(s) may be trapped between the sensor housing and the electrode. The body 3 is provided with a suitable gas diffusion barrier in the centre of the region covered by the electrode 4, in this case a capillary with diameter 0.8mm. Both the body 3 and a backplate 9 are injection moulded from the thermoplastic ABS material `Cevian V660'.

Three current collector wires 1a, 1b, 1c and grommet 2a, 2b, 2c assemblies of the type shown in Figure 1 are inserted into the body 3 through receiving apertures 12a, 12b, 12c designed to provide an interference fit, so that the current collectors protrude through apertures 12a, 12b and 12c. They are pressed home into receiving apertures in the body 3 before attaching gold-plated phosphor bronze clips 8 which locate on and are retained by barbs on the housing, thereby trapping the current collector wires and providing electrical contact to external circuitry. The clips 8 also provide a degree of lateral compression thereby increasing the sealing force between the grommet, body and current collector to the required level. (In the example shown, these processes have been performed manually, although the relevant components have been designed to allow for the possibility of automated insertion.) If preferred, the section of wire 1 which protrudes through the grommet 2 on the outside of the sensor body 3 may be spot welded to clip 8 before attachment to provide greater robustness in the connection. The current collectors for the reference and counter electrodes are then bent up into a vertical direction, allowing the remainder of the electrode stack to be assembled in a conventional interleaving fashion.

A porous glass wool separator 5 (12mm diameter) is placed over the sensing electrode 4, so trapping the sensing current collector between the two. A 6.7mm diameter reference electrode 6 (fabricated in an analogous way to the sensing electrode 4) is then placed in the centre of separator 5 (again with the electrocatalyst facing upwards in the Figure). The associated current collector is bent down from its vertical position to make contact therewith, before a second 12mm diameter separator 13 is laid on top, trapping the current collector in place. The third (counter) current collector is then brought down to a horizontal position before a conventional gas diffusion annular counter electrode 7 (internal/external diameters 6.7/11.5mm) completes the stack. The sensor backplate 9 is then located into a mating recess in body 3 before being ultrasonically welded into position, thereby achieving a liquid-tight seal and producing the required degree of compression on the electrode stack to give good electrical contact throughout the assembly. Electrolyte (0.15ml of 5M sulphuric acid) is then injected into the housing through a filler port 11 which is then closed by heatstaking. After attaching a gas permeable bulk flow membrane 10 to the front of the body 3, the sensor is then tested and prepared for dispatch to the customer.

Figure 3 shows a cross sectional detail of a grommet/current collector seal area after closure of the casing. The electrode stack in the region where the current collector 1 exits aperture 12 in sensor body 3 has been omitted for clarity. The lateral compression provided by clip 8, together with the radial compression provided by insertion of grommet 2 into aperture 12 provides the long sealing path between the current collector, sensor body and grommet as shown.

The completed dimensions of the sensor are 17mm wide by 17mm deep by 4mm high which is extremely compact in comparison with most commercial devices of comparable performance. This size reduction is, to a great extent,

facilitated by a series of design choices which rely on the robust electrolyte seal as described by this invention.

In the examples described so far, a single grommet has been threaded onto the wire 1 as shown, for example, in Figure 1(b). For ease of manufacture, however, it is preferred to injection mould a series of grommets 2 onto a wire 1 as shown in Figures 4 and 5. The wire 1 can then be cut at suitable positions so as to produce a series of grommet/wire units for assembly into the sensor.

Figure 6 illustrates a second example of a sensor in which the seal has been fabricated by the backfilling technique. The body 3, current collector 1 and clip 8 are all constructed (and perform the same functions) as previously described. However, in this case the current collector 1 is initially held in position by means 30. This can be achieved by moulding a short section of the current collector in to the main body, as shown here. The contact between current collector 1 and body 3 in the region of 30 does not necessarily form part of the electrolyte seal. Thus, the fact that body 3 is made from a relatively noncompliant material is not a drawback. Indeed, the characteristics of the material used to fabricate the body 3 in this case are to some extent dictated by the requirement that it should be able to withstand the temperatures and pressures which are generated during the backfilling process. Typical examples of materials which can be used in these applications are ABS and similar polymers for the body 3 and thermoplastic elastomers for the backfill 20.

Alternatively, 30 may comprise a separate clip or other current collector locating means. In either case, the purposes of 30 are:

(a) to retain current collector 1 in position whilst the backfilling process takes place and

(b) to seal the inner part of the seal cavity so preventing leakage of the backfilling material into the interior of the body.

Standard 2 shot injection moulding technology can be used to facilitate the backfilling in a single tool. Alternatively, the body/current collector assembly can be formed in a first tool and then transferred to a second tool where the backfilling process occurs. In either case, material 20 is injected into the cavity around current collector 1 and finally clip 8 is attached to complete the assembly.

CLAIMS

1. An electrochemical gas sensor having a housing in which is located sensing and counter electrodes in contact with a liquid electrolyte; and current collectors in contact with respective electrodes and extending through respective apertures in the housing, each aperture including a compliant seal which is resiliently urged against the current collector and the wall of the aperture to seal against the passage of electrolyte through the aperture.

2. A sensor according to claim 1, wherein the compliant seal comprises an elastomeric grommet.

3. A sensor according to claim 2, wherein the grommet has one or more ridges extending around its outer circumference.

4. A sensor according to claim 2 or claim 3, wherein the length of the grommet is large with respect to its outer diameter.

5. A sensor according to claim 4, wherein the length of the grommet is more than twice its outer diameter.

6. A sensor according to claim 4 or claim 5, wherein the length of the grommet is more than 20 times its inner diameter.

7. A sensor according to any of claims 2 to 6, wherein the grommet is an interference fit in the aperture.

8. A sensor according to any of claims 2 to 7, wherein the grommet has a lateral extension extending beyond the housing wall, a clip being mounted on the lateral extension to compress it, the clip contacting the current collector.

9. An electrochemical gas sensor current collector carrying an elastomeric grommet.

10. A current collector according to claim 9, wherein the grommet is a tight fit on the current collector.

11. A method of providing an elastomeric grommet on an electrochemical gas sensor current collector, the method comprising moulding, for example injection moulding, the

grommet about the current collector.

12. A method according to claim 11, comprising moulding a plurality of grommets onto the current collector at spaced positions along its length.

13. A method according to claim 12, wherein the plurality of grommets are formed by locating the current collector in a mould, moulding a grommet about the current collector using the mould, indexing the current collector and moulded grommet through the mould, and then repeating the moulding process.

14. A method according to claim 12 or claim 13, further comprising cutting the current collector into a number of individual current collector/grommet elements.

15. A method of sealing a current collector in an aperture of an electrochemical gas sensor housing, the method comprising threading a current collector according to claim 9 or claim 10 through the aperture and push fitting the grommet into the aperture.

16. A method of sealing a current collector in an aperture of an electrochemical gas sensor housing, the method comprising locating the current collector in the aperture; and filling the aperture with a compliant sealing material.

17. A method according to claim 16, wherein the compliant material comprise a thermoplastic elastomer.

18. A method according to claim 15 or claim 16, wherein the step of locating the current collector in the housing wall aperture comprises moulding, for example injection moulding, the housing about the current collector.